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DR. SCHULMAN-RNP

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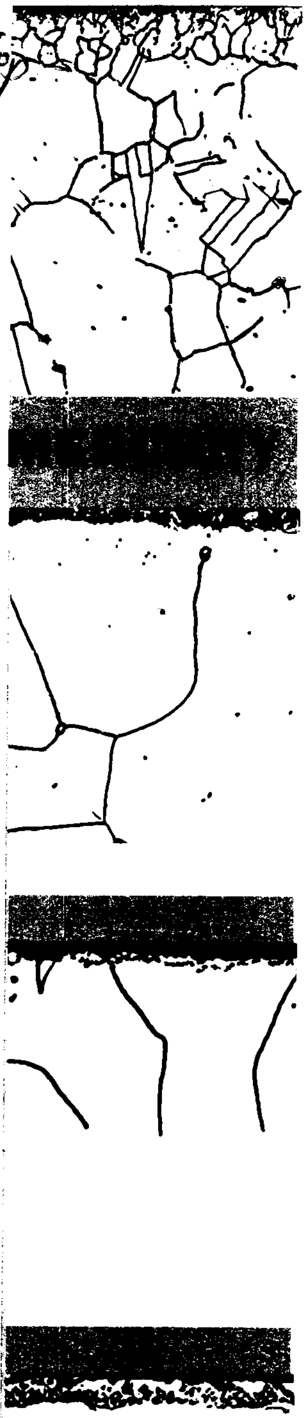

CORROSION LOOP TESTING PROGRAM

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Report No. 0584-04-4 (Quarterly)

October 1962

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Aerojet-General CORPORATION • AZUSA, CALIFORNIA

A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

26 October 1962

Report No. 0584-04-4

MERCURY CORROSION LOOP TESTING PROGRAM

Contract NAS 3-1925

No. of Pages: 30

Period Covered: 1 July through 30 September 1962

AEROJET-GENERAL CORPORATION

Azusa, California

NASA CR-50,342

,72 23

ABSTRACT

This report describes the fabrication, testing, and analysis work accomplished during the fourth quarter (July - September 1962) of the Dynamic Mercury Corrosion Loop Testing Program. Operation and modification of the first of the ten loops were continued. Some of the special alloys to be used in the later loops were received.

CONTENTS

CONTRACT FULFILLMENT STATEMENT

Work at Aerojet has been authorized under NASA Contract NAS 3-1925. Testing is being conducted at Aerojet-General Nucleonics (an Aerojet subsidiary), San Ramon, California, under Subcontract No. OP328862. This is the fourth in a series of quarterly reports submitted in partial fulfillment of Contract NAS 3-1925.

I. INTRODUCTION

Several power conversion systems employing mercury boiling and condensing cycles are under development within the aerospace industry. These systems share a common need for quantitative information about the long-term compatibility of mercury and mercury-containment materials.

The Mercury Corrosion Loop Testing Program currently being conducted by Aerojet-General Corporation is designed to provide a substantial amount of this information. The specific objective of the program is to obtain sufficient experimental data to provide a firm basis for selection of a suitable mercury-containment material. The material must be able to withstand a nominal mercury boiling temperature of 1075°F for an operational life of 10,000 hours. Several alloys, including Haynes 25, will be tested in ten forced-convection test loops. Testing periods will exceed 2,000 hours and, on some loops, will approach 10,000 hours. The loops will duplicate as closely as possible the cycle conditions and flow characteristics of the SNAP-8 Power Conversion System.

In addition, the loop data will be used to estimate the location and degree of corrosion attack that may be expected to occur in the operational system.

The materials philosophy for the SNAP-8 program is currently undergoing a re-examination in the light of recent corrosion data. Consequently, the objectives of the Corrosion Loop Program have been revised, and are now as follows:

A. Operate Loops 1 and 2 (Haynes 25 construction), and to make necessary revisions to achieve stable flows, pressure, and design conditions.

B. Evaluate the co-extruded Cb-316SS tubing.

C. Plan a revised loop program, with the emphasis on columbium as the referenced material.

II. SUMMARY OF ACCOMPLISHMENTS - 1 JULY THROUGH 30 SEPTEMBER 1962

The intermittent operation of Loop No. 1 continued. A total operating time of 847 hours has been accumulated.

Minor design changes were made in Loop No. 1, and the flow stability was greatly improved.

Four back vanes were added to the centrifugal pump, and performance was improved until the pump nearly meets design requirements.

The co-extruded and the co-drawn 9Cr-1Mo-316SS* tubing was received from the vendors. The co-extruded tubing exhibited a strong bond while the co-drawn tubing had only a mechanical bond.

III. DISCUSSION

A. SPECIAL ALLOY PROCUREMENT

The co-drawn and co-extruded 9Cr-1Mo 316SS tubing was received from the vendor. The co-extruded Cb-316SS tubing was shipped by the vendor on 10 September. Procurement of other alloys for use in valves and miscellaneous loop hardware was suspended because of the impending redirection of the program.

B. ALLOY EVALUATIONS

Approximately 150 ft of co-extruded 3/4-in.-dia tubing (9Cr-1Mo alloy clad with 316 stainless steel) fabricated by Nuclear Metals is being evaluated for formability, bond integrity, and heat-treatment effects. The duplex tubing, as measured at random lengths, has an inside diameter of 0.652 in. with a 0.050-in. wall. The inside portion of the tubing wall (9Cr-1Mo) has an average thickness of 0.020 in. and the outer, Type 316SS portion has an average thickness of 0.030 in. The tubing was manufactured by a co-extrusion process, cold-drawn to size, and annealed.

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*The following abbreviations are commonly used in reports of this program:

316SS = Type 316 stainless steel
9Cr-1Mo = 9 chromium - 1 molybdenum steel alloy
AM-350 = AM-350 precipitation-hardened stainless steel.

To establish the formability of the tubing, an 18-in. length was cold-formed smoothly with a pipe bender into 90° bends over a 3-in. radius. Each bend was satisfactorily made both with and without the aid of a low melting filler metal inside the tubing (Figure 1). Visual and metallographic examinations disclosed no ruptures, buckling or interface separations at the cladding interfaces of the bent regions. Pre- and post-bend micrometer measurements showed some ovality; the tube bends with and without filler were 0.005 and 0.015 in. out of round, respectively. The microstructure of the as-received (annealed) material revealed no apparent metallurgical bond (Figure 2). Such a bond would normally be observed as a diffusion layer. However, several preliminary tensile shear tests indicated the bond was as strong as either of the duplex alloys. According to the test report from Nuclear Metals, chisel tests indicated that an inseparable metallurgical bond had been obtained.

Microstructural effects due to thermal cycling between 1400 to 400°F, or to heat treatments at 1300 and 1475°F for 100 hours are shown in Figures 3 through 5. Except for a slight increase in grain size of the 9Cr-1Mo alloy at the bond interface, no diffusion was observed after 100 thermal cycles (Figure 3)* or 100 hours heat treatment at a temperature of 1300°F (Figure 4). The heat treatment at 1475°F for 100 hours produced extreme grain growth, with some diffusion at the joining interfaces (Figure 5).

C. LOOP FABRICATION

The principal task during this report period was to operate Loop No. 1 and to make indicated design revisions to promote stable operation with minimum flow and pressure fluctuations.

Three principal design changes were made in the loop:

1. The turbine simulator, as illustrated in the previous quarterly report, was installed in the loop.

2. A bellows-sealed valve was located within 18 in. of the boiler inlet to serve as a variable pressure restriction.

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*Thermal cycles: 4 min held in furnace, and 2 min cooled in air.

3. A 4-ft length of 3-in. pipe was installed between the boiler discharge and the turbine simulator to serve as a quieting chamber.

Fabrication continued on the second Haynes 25 loop and the original schedule was changed to accommodate the above mentioned design changes. Fabrication of six 9Cr-1Mo centrifugal pumps and six 9Cr-1Mo boilers and special valves was suspended on instructions from the NASA Project Officer. The existing Haynes 25 centrifugal pumps were modified to incorporate four additional back vanes on the impeller. Because the centrifugal pumps exhibited poor net positive suction head (NPSH) characteristics, a jet booster pump was designed and fabricated. The disassembled pump is shown in Figure 6. By mutual agreement between Aerojet and the NASA Project Officer, it was decided to omit, at least temporarily, the erosion experiment from the turbine simulator in Loop No. 2. Details of the erosion experiment were presented in the last quarterly report.

D. LOOP OPERATION

During the quarter, Loop No. 1 was operated four times for a total operation time of 464 hours. Details of runs 7 through 10 are presented in Table 1.

During July, Loop No. 1 was operated for 417 hours, bringing the total accumulated hours of operation to 800. A significant improvement in boiler heat transfer was observed during this period. At rated flow (1200 lb/hr) and pressure (275 psia), mercury quality went from 50 to 100%. Boiler power changed from 27 to 50 kw. The loop was shut down because flow was restricted by a significant buildup of corrosion products in the turbine-simulator nozzles. A detailed review follows.

Prior to startup on 5 July, shields were installed in the radiator. These consisted of two aluminum panels suspended between the radiator tubes and the Platecoil cooling panels. The shields are movable vertically and provide a means of controlling the outlet temperature of the mercury from the radiator.

The original Haynes 25 pump was removed from the loop and replaced with one with a reduced front vane diameter. Previous testing had shown that this pump required more NPSH than the full-vane unit. To provide a continuous check on the pump, a pressure transmitter-recorder system was connected to the pump outlet.

The loop was started on 5 July, and operated for 151 hours. The mercury flow rate was approximately 1200 lb/hr with an exit vapor quality of 50%. The boiler pressure was 275 psia. The run was terminated when the bearings in the pump failed. During the run, continuous attention was required to maintain flow through the loop by regulating pressure in the pressurizer. It was found that the pump outlet pressure could not be held steady with the mercury level in the pump cavity below the high probe. To minimize mercury contamination of the bearings, the pressurizer pressure was alternately reduced and increased to hold the output pressure of the pump high enough to maintain flow, and to operate the pump with an intermittent high level of mercury in the cavity.

After termination of the run, the bearings and seal were replaced. The loop was started again on 12 July, and operated for 183.5 hours. Operation was again hampered by the poor NPSH characteristics of the pump. However, wetting of the Haynes 25 tubing apparently occurred during the run. The wetting of the tubing was indicated by a significant increase in the power requirements to the boiler. At mercury flow rates of 1200 to 1400 lb/hr, vapor qualities of 60 to 70% were calculated; at flow rates of 800 to 900 lb/hr, vapor qualities of 90 to 100% were calculated.

The run was terminated to allow replacement of the hand-operated turbine simulator valve with an air-operated valve of larger capacity. A hand-operated bellows-sealed valve was installed in the inlet line to the boiler. This latter change was made to provide more stability in the loop flow characteristics. It has been observed in this loop that flow is unstable, with a variation of ±25% in a 1/2-sec period.

The loop was started on 27 July and operated for 82.5 hours. Mercury flow at the start of the run was 1300 lb/hr with a quality of 100%. Pressure at the boiler outlet was 275 psig. As the run progressed, a gradual increase in the boiler pressure was noted. The flow and the boiler temperature were reduced to hold the boiler pressure below 350 psig. At the termination of the run, the NaK temperature in the boiler was 1210°F and the mercury flow rate had been reduced to 750 lb/hr. Analysis of loop characteristics indicated that the pressure increase and reduced flow were caused by a restriction in the turbine simulator. Examination revealed a significant buildup of corrosion products in the nozzles of the turbine simulator. The analysis of these is presented in Section III,E, below.

There was no loop operation during August, except for testing in the pump test loop. The test cell for Loop No. 1 was cleaned, and provisions were made for installing the expansion chamber and the turbine simulator.

On 25 September, the loop was evacuated to 10 μ* and filled with mercury. Liquid remaining from the July test run was left in the boiler coil to minimize oxidation of the Haynes 25, and to facilitate wetted heat transfer. When the mercury pump was started, argon was inadvertently introduced into the loop because of a short-circuited level probe in the pump. The loop was drained, evacuated, and filled with mercury. The loop was re-started on 26 September and in 3 hours the boiler was operating at 80% quality. At rated flow of 1200 lb/hr, the boiler pressure of 275 psia was maintained by the turbine simulator. A total boiling time of 48 hours was obtained when a plant power failure caused the loop control system to shut down. A re-start was attempted, but argon had entered the loop through the pump seal cavity.

During the operation period, the exit vapor quality varied between 80 and 95%. Saturated vapor could not be obtained even at reduced flow rates. The heat transfer rates were approximately 5% less than those experienced in the two previous runs. The reduction is attributed to a superficial oxidation of the Haynes 25 boiler coil after a 5-week exposure to air. It is anticipated that prolonged operation at design temperature will produce saturated vapor.

Pressure and flow fluctuations were observed during the loop operation. It was found that throttling the boiler inlet stream through the manual valve aided the loop stability. When the 150-psi pressure drop was expended, the flow fluctuations were ±1.5%, and the inlet pressure varied ±3.5 psi from the 250-psig

*Measured between vacuum pump and liquid nitrogen cold trap.

nominal pressure. Both variations occurred with a period of 0.6 sec. The boiler outlet pressure appeared stable with a slight pressure variation of ±1 psi over a period of 2 sec. These values are a considerable improvement over the previous behavior, where the flow fluctuated ±25%.

Further testing is planned using different entrance pressure drops so that the minimum stabilizing ΔP can be found. The intent is to eventually replace the manual valve with an orifice.

Presently, the loop is shut down to permit reduction of the cooling capability of the radiator and replacement of the turbine simulator which had developed cracks in the Haynes 25 tubing. A second turbine simulator received from the vendor also leaks. A third unit is being fabricated by Aerojet-General Nucleonics.

Considerable centrifugal pump testing was conducted to obtain a hydraulically stable pump. It was determined that variations in the back vane and front vane clearances affected the pump suction head, discharge head, and horsepower. After a systematic variation of these clearances, it was found that a pump with eight back vanes and four front vanes produced the following results:

Front vane clearance	0.055 in.
Back vane clearance	0.040 in.
NPSH	16 psia
Shutoff head	500 psia
Horsepower	2.9

One Haynes 25 pump was cleaned, seal welded, and installed in Loop 1.

E. LOOP EVALUATION

The evaluation of the turbine simulator which restricted normal mercury flow during operation of Loop No. 1 is approximately 90% complete. As shown in Figure 7, three nozzles are incorporated in the simulator. Each of the nozzles has a different orifice geometry, and variation in orifice sizes ranges from 0.202 in. for No. 1 nozzle (item 5) where mercury enters the simulator, to 0.299 in. for No. 3 nozzle (item 12) where mercury exists.

According to its operational history, this simulator was operated at temperatures of about 1080°F, with mercury flow rates for various operational cycles ranging from 300 to 1400 lb/hr. The total time during eight cycles of operation amounted to ~800 hours before failure of the simulator.

Visual examination showed corrosion products in the orifices of all nozzles. About 90% of the corrosion products were found in No. 1 (inlet) nozzle (Figures 8, 9 and 10). The diameter of the No. 1 nozzle hole was found to be 0.107 in., indicating an opening only 25% of the normal size.

Mercury was distilled from the corrosion products and the residue was spectrographically analyzed for elements. The major elements found were as follows:

Co - Principal
Fe - 4.7%
Cr - 3.4%
W - 2.0%

The elements found are basic constituents of Haynes 25 and of portions of the loop components and the nozzles.

Metallographic examination of sections from No. 1 nozzle showed that the inside surface had eroded ~ 3 mils in depth. This was primarily noted at the grain boundaries, as shown in Figure 11. At higher magnifications an unknown phase formation was noted in the grain boundaries (2 mils in depth) nearest the eroded surface. Beyond a total depth of 5 mils from the surface the unknown boundary phase disappears. Investigations and evaluations are being continued.

Examination showed that bearing failure caused failure of the Haynes 25 pump during operation of Loop No. 1. The possible contributors to the bearing failure were liquid mercury in the bearing lubricant, and thrust loading on the lower bearing.

Mass transfer deposits were found on all of the pump parts and corrosion products were floating on the mercury trapped in the pump at disassembly. The major elements in these deposits and in the particulate were Cr, Ni, Fe and Co.

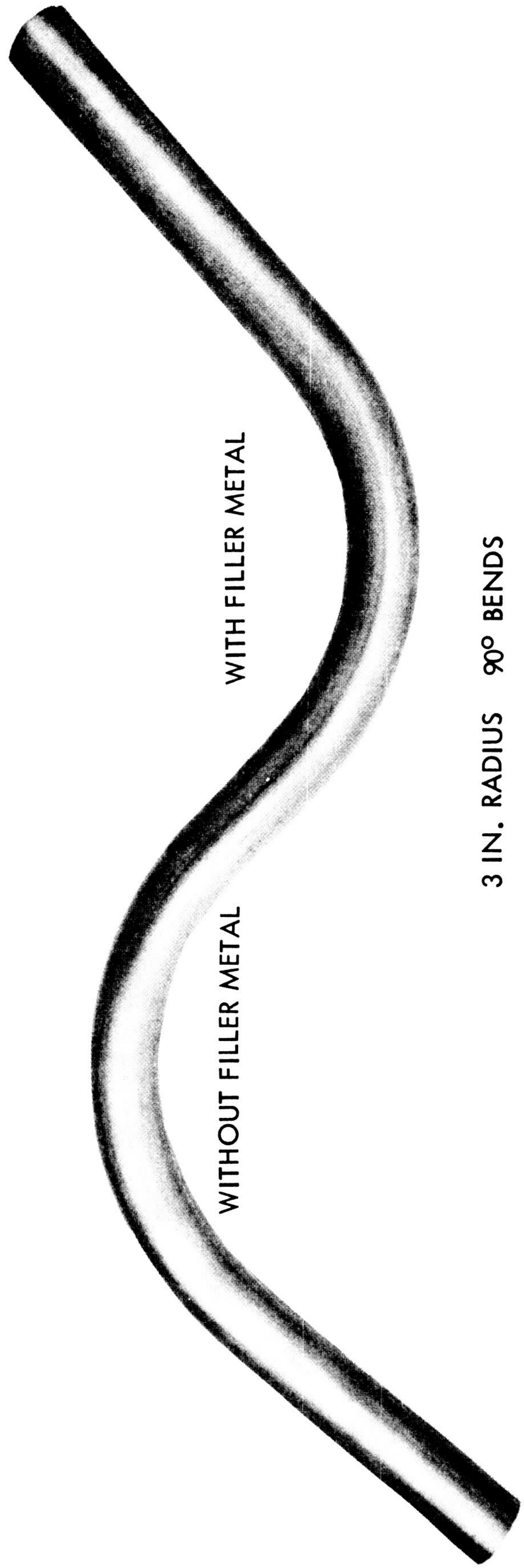
IV. WORK PLANNED FOR NEXT QUARTER

Operation of Loop No. 1 will continue in order to obtain saturated vapor, and for further experiments to determine the practical boiler inlet stabilizing pressure drop. Operation of Loop No. 2 will be started. The co-extruded Cb-316SS tubing will be received and evaluated. Planning of the redirected loop testing program will be completed, and the program will be submitted to NASA for approval.

TABLE 1

OPERATION DATA, LOOP NO. 1 - HAYNES 25

Run No.	Start	Duration, Hours	Approximate Flow Rate lb/hr	Boiler Outlet Pressure, psig	Vapor Quality	Remarks
During Interim Period:			Installed aluminum baffles in radiator section; cut out old pump and replaced with new Haynes 25 pump with reduced impeller diameter. Installed pressure recorder on pump outlet; removed bypass from pump cavity to pressurizer.			
7	July 5	151	1200	265	50%	Pump would not put out 400 psig with red light off. Flow control varied as it is dependent on pump output which was directly related to pressurizer pressure.
During Interim Period:			Replaced bearings and seal in pump; replaced rupture disc on pressurizer.			
8	July 12	183.5	1200-1400 800-900		60-70% 90-100%	Continuous adjustment of pressurizer pressure is required to maintain mercury level in pump.
During Interim Period:			Replaced bearing and seal (Seal looks good. Upper bearing wet with Hg: lower bearing grease extruding and some Hg in bearing). Installed valve on boiler inlet; replaced T/S Hoke valve with air operated Flow Systems valve to reduce pressure drop. Changed most of 1/4 in. Cu lines on T/S distilled water to 1/2 in. Cu to reduce pressure drop.			
9	July 27	82.5	1300-1200 750		100% 100%	Heat balance indicates that superheat should be obtained even though the outlet temperature does not rise. Boiler pressure gradually increased indicating flow restriction between boiler and radiator.
During Interim Period:			Added a pressure tap between T/S valve and radiator. Installed pump with 8 back vanes and full impeller diameter. The turbine simulator was replaced with one having a single sonic nozzle. The top center Platecoil was disconnected.			
10	Sept. 26	47	1200	260	80-95%	The sonic nozzle maintained the boiler pressure at rated flow.

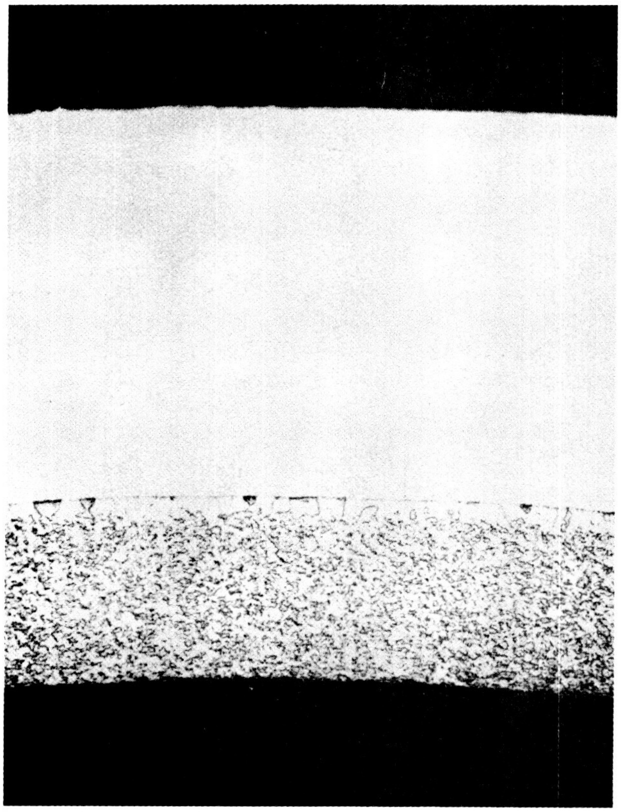


3 IN. RADIUS 90° BENDS

DUPLEX TUBE – 9 Cr-1 Mo CLAD WITH 316 STAINLESS STEEL
FORMABILITY BENDS

FIGURE 1

PHOTO MICROGRAPHS OF THE 9 Cr - 1 Mo TUBING
CLAD WITH 316 STAINLESS STEEL

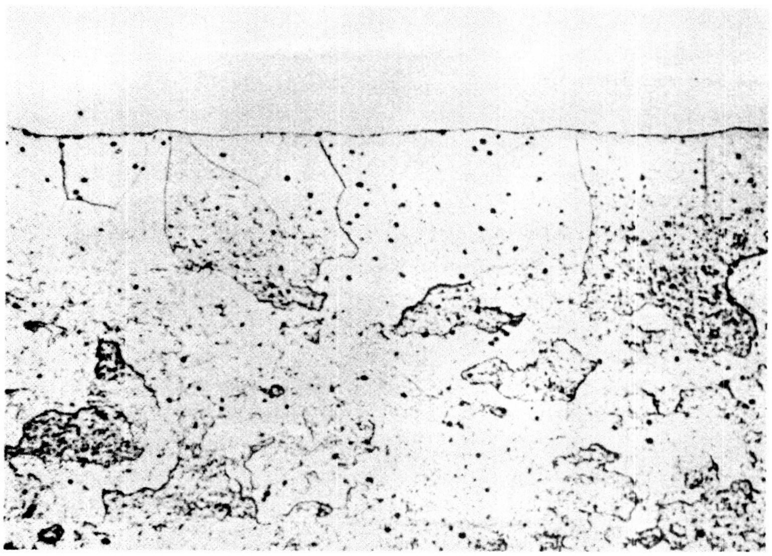


316 STAINLESS STEEL

9 Cr - 1 Mo STEEL ALLOY

2449 60X

AS RECEIVED (DRAWN AND ANNEALED)
ETCHED: GLYCERA REGIA



316 STAINLESS STEEL

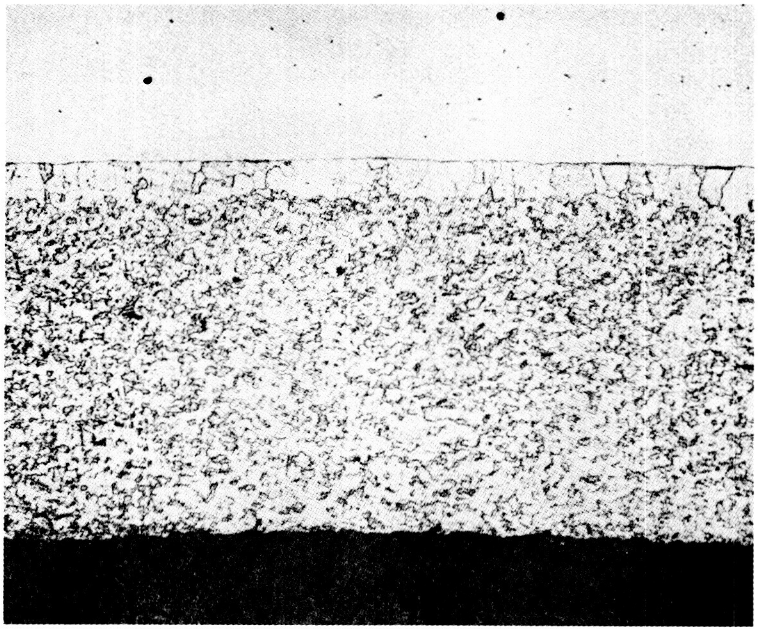
9 Cr - 1 Mo STEEL ALLOY

2450 500X

AS RECEIVED (DRAWN AND ANNEALED)
ETCHED: GLYCERA REGIA

FIGURE 2

9 Cr - 1 Mo TUBE CLAD WITH 316 STAINLESS STEEL
AFTER 100 THERMAL CYCLES 400° - 1400°F

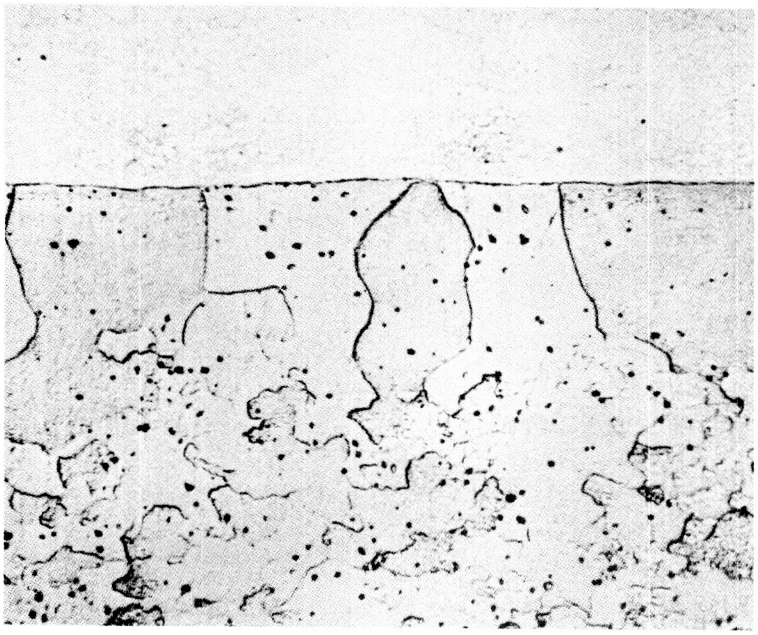


316 STAINLESS STEEL

9 Cr - 1 Mo STEEL ALLOY

2482 100X

ETCHED: GLYCERA REGIA



316 STAINLESS STEEL

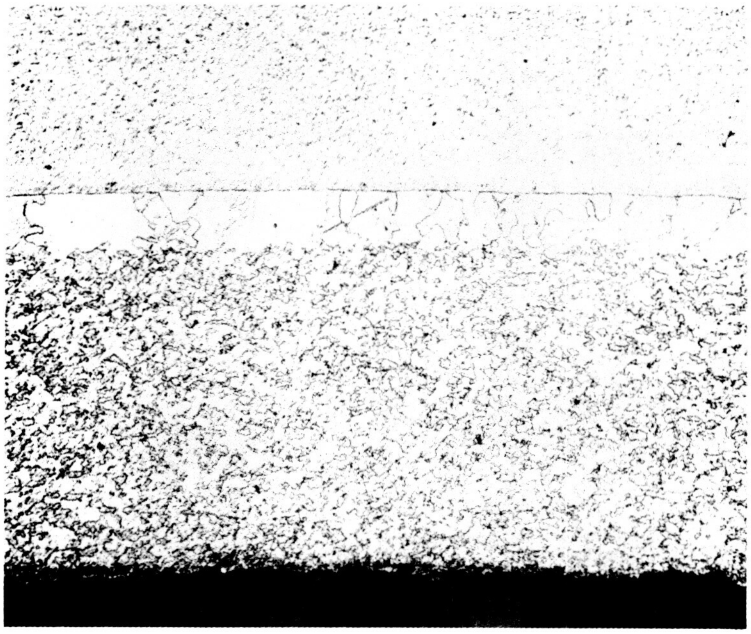
9 Cr - 1 Mo STEEL ALLOY

2481 500X

ETCHED: GLYCERA REGIA

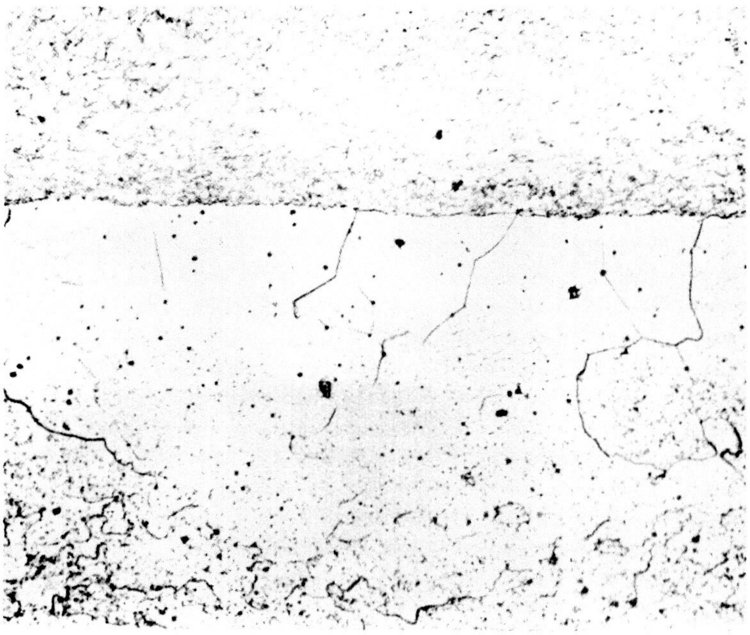
FIGURE 3

9 Cr - 1 Mo TUBE CLAD WITH 316 STAINLESS STEEL
HEAT TREATED @ 1300°F FOR 100 HR



2488 100X

ETCHED: GLYCERA REGIA

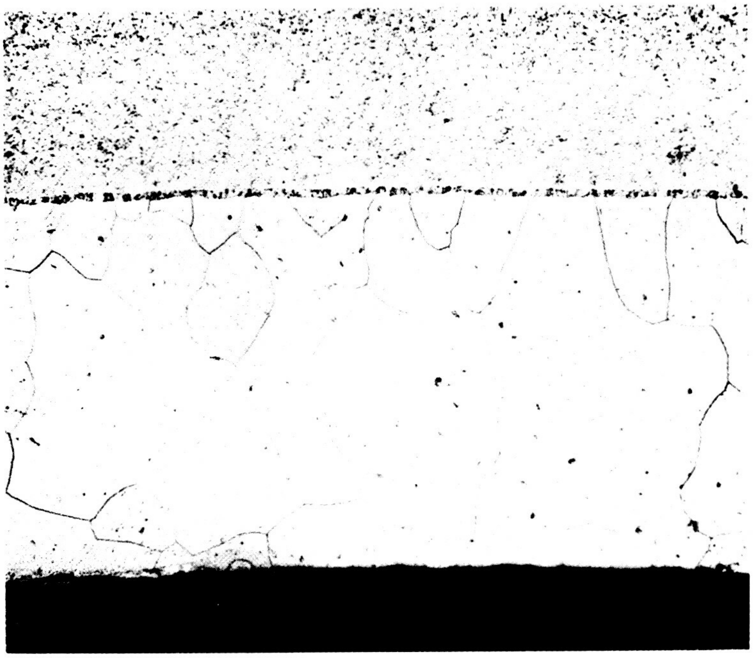


2487 500X

ETCHED: GLYCERA REGIA

FIGURE 4

9 Cr - 1 Mo TUBE CLAD WITH 316 STAINLESS STEEL
HEAT TREATED @ 1475°F FOR 100 HR



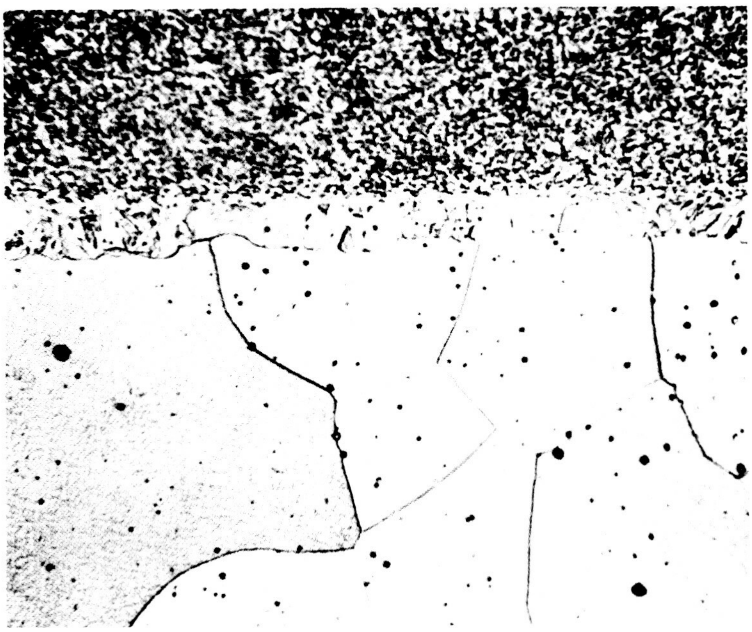
316 STAINLESS STEEL

DIFFUSION ZONE

9 Cr - 1 Mo STEEL ALLOY

2490 100X

ETCHED: GLYCERA REGIA



316 STAINLESS STEEL

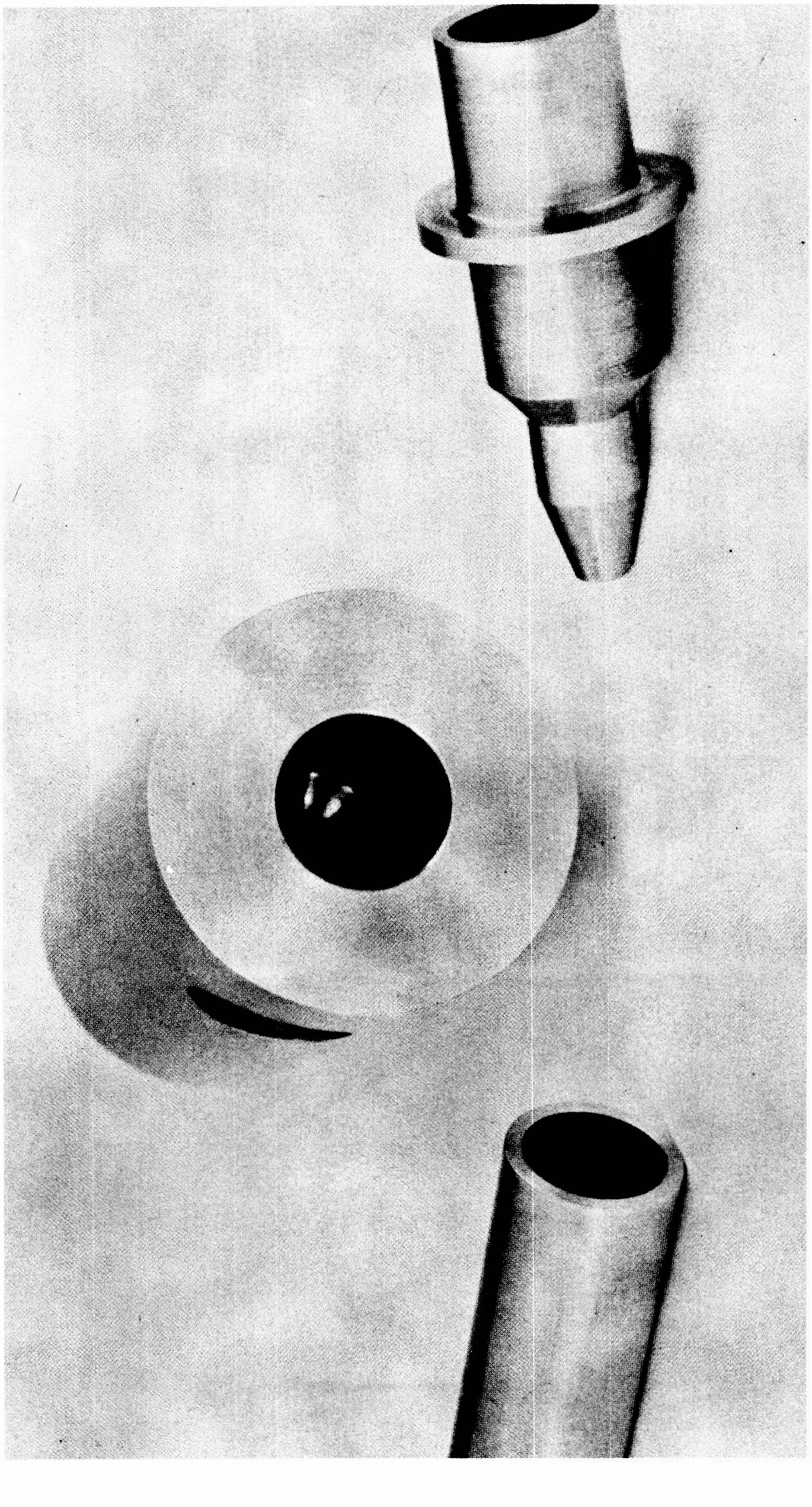
DIFFUSION ZONE

9 Cr - 1 Mo STEEL ALLOY

2489 500X

ETCHED: GLYCERA REGIA

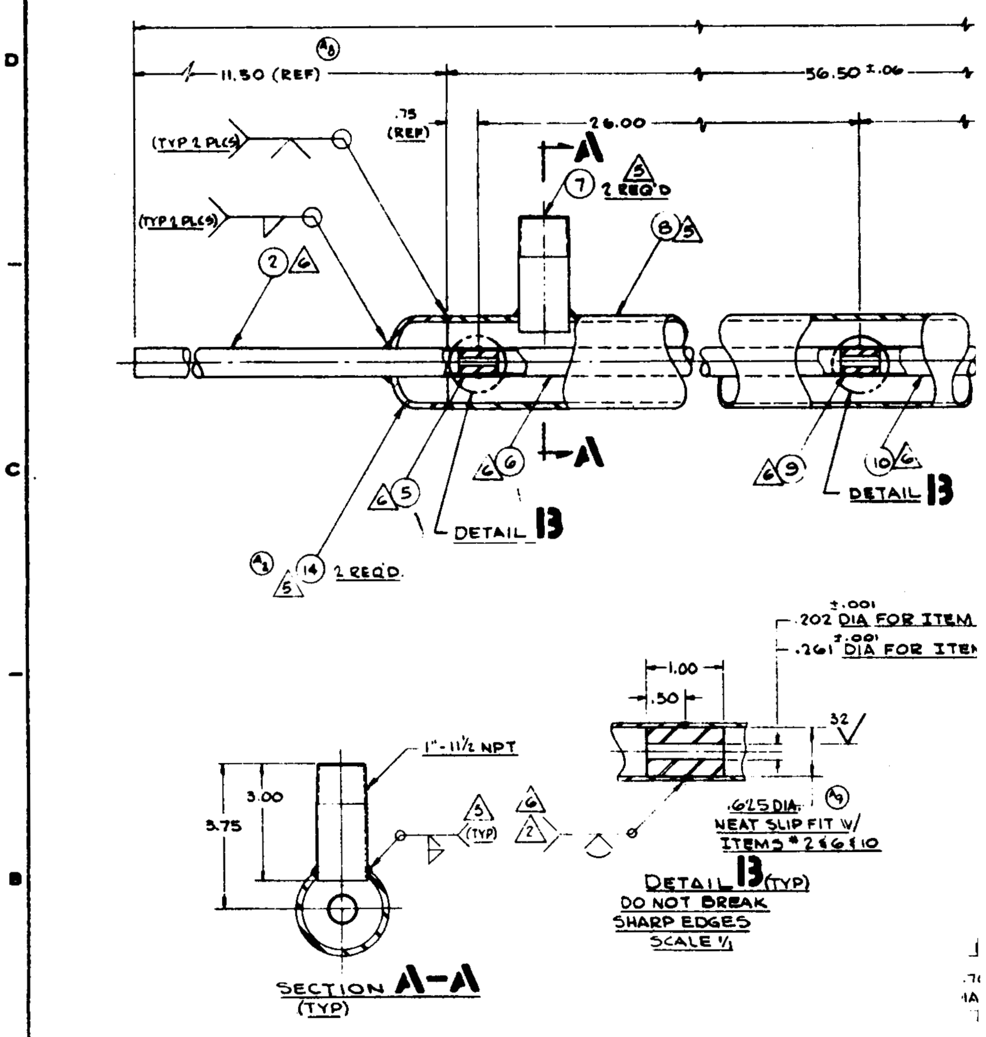
FIGURE 5



JET BOOSTER PUMP COMPONENTS

FIGURE 6

4



7. ALL WELDING NEEDED TO MAKE REQ'D LENGTHS OF HAYNES 25 TUBING. TO BE BUTT WELDED AND 100% X-RAY INSPECTED. NO DEFECTS ALLOWED.

A7 6 HELIUM LEAK CHECK ASS'Y OF ITEMS 2, 5, 6, 9, 10, 12 & 13 (MERCURY PRESSURE DROP
@ 600 PSI, HELIUM LEAK RATE NO GREATER THAN 5×10^{-10} CC/SEC AT 600 PSI AT ANY

A4 5 HYDROSTATIC TEST ITEMS 7, 8 & 14 (COOLING WATER JACKET) LEAK PROOF @ 100 PSI FOR 30 MIN.

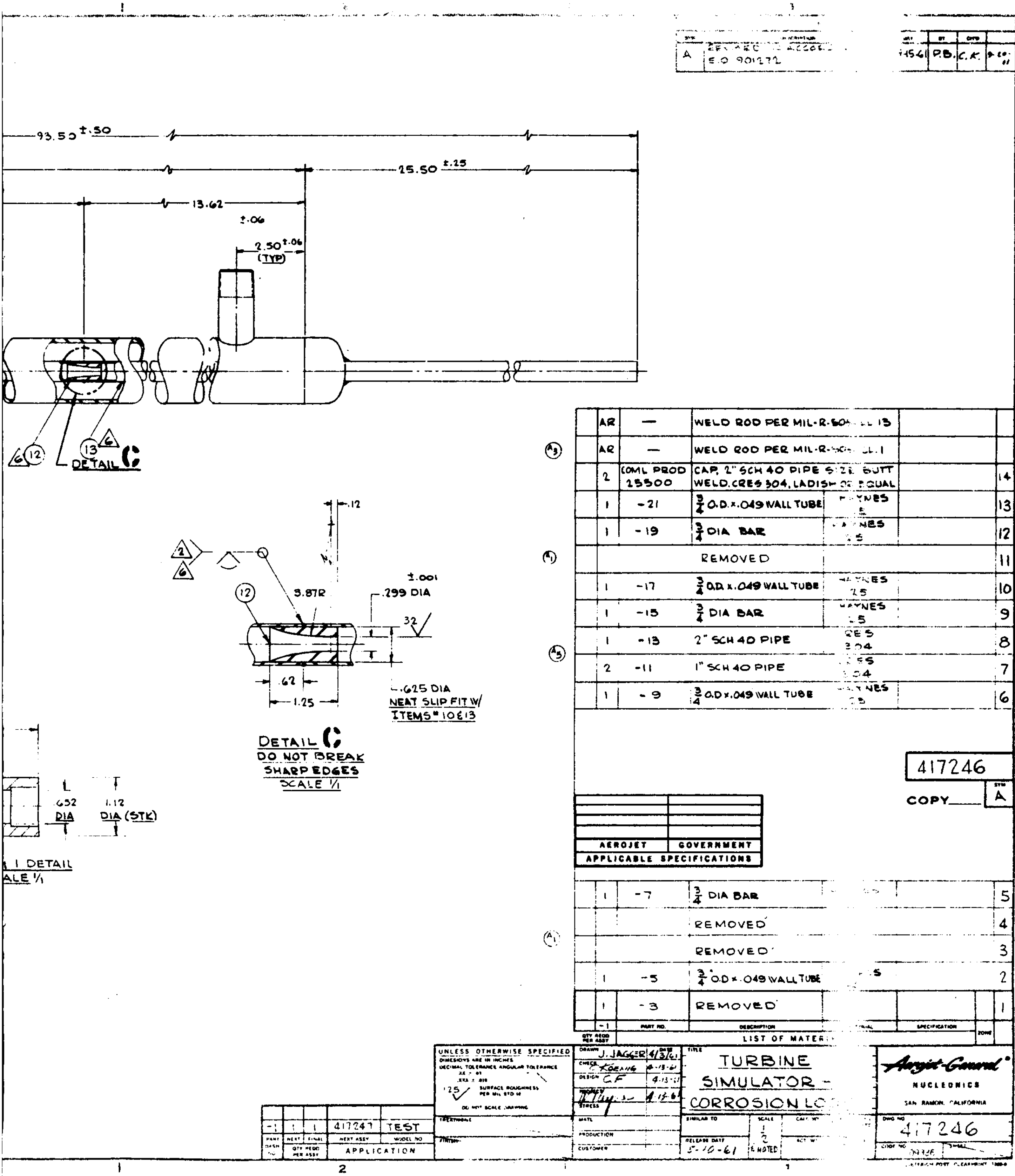
4 REMOVED A6

3. TAG WITH PART No.

2 WELDS MARKED 2 TO BE 100% ROOT PENATRATION.

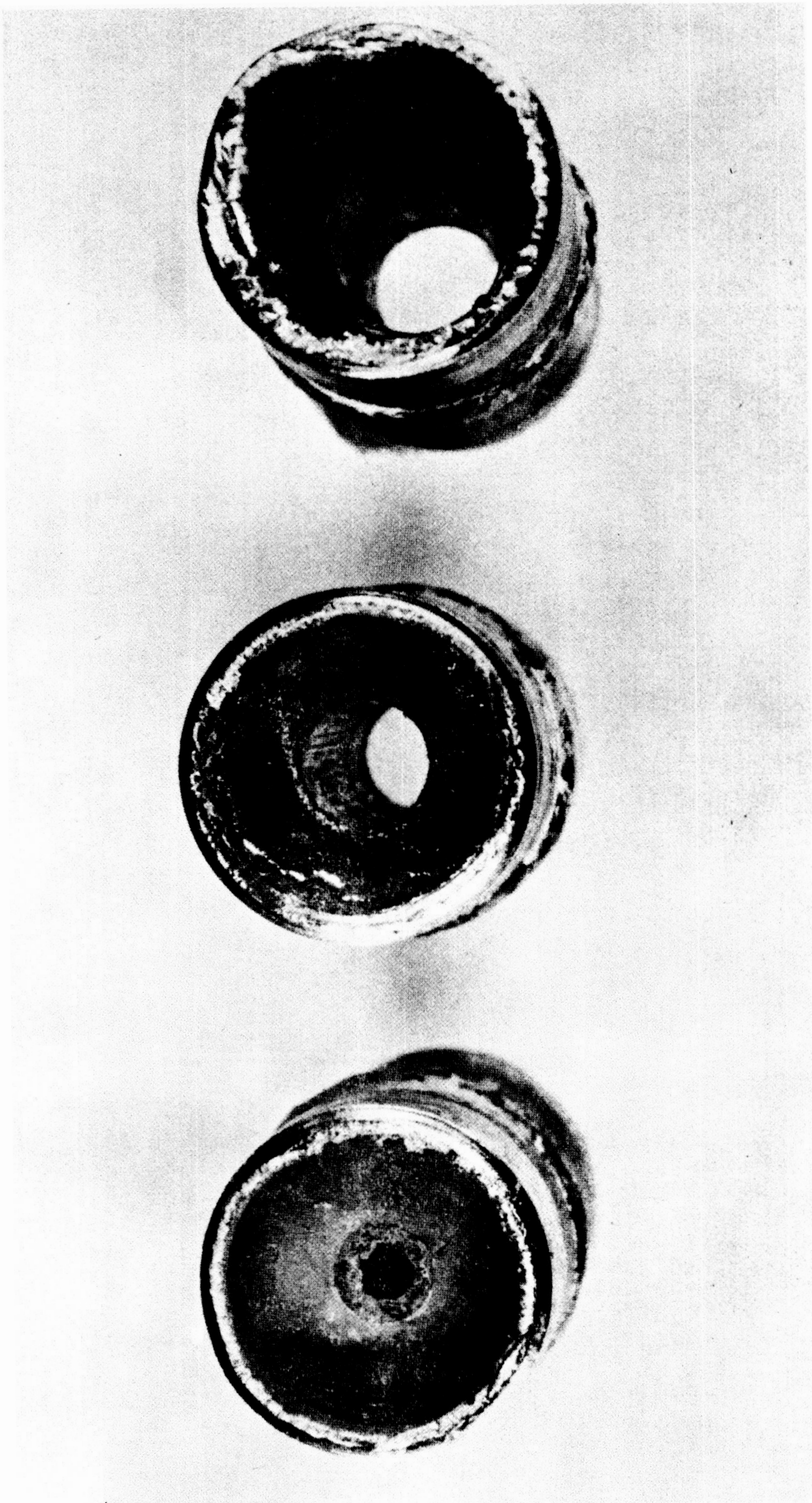
NOTES. 1. REMOVE ALL BURRS AND SHARP EDGES, EXCEPT AS NOTED.

4



93.50 ±.50
25.50 ±.25
13.62 ±.06
2.50 ±.06 (TYP)
DETAIL C
.12
3.87R
±.001 .299 DIA
32
.62
1.25
.625 DIA NEAT SLIP FIT W/ ITEMS # 10 & 13
DETAIL C
DO NOT BREAK SHARP EDGES
SCALE 1/1
.652 DIA
1.12 DIA (STK)
1 DETAIL
ALE 1/1
AR — WELD ROD PER MIL-R-
AR — WELD ROD PER MIL-R-
2 COML PROD 25500 CAP, 2" SCH 40 PIPE SIZE BUTT WELD, CRES 304, LADISH OR EQUAL 14
1 -21 3/4 O.D. x .049 WALL TUBE HAYNES 13
1 -19 3/4 DIA BAR HAYNES 12
REMOVED 11
1 -17 3/4 O.D. x .049 WALL TUBE HAYNES 10
1 -15 3/4 DIA BAR HAYNES 9
1 -13 2" SCH 40 PIPE CRES 304 8
2 -11 1" SCH 40 PIPE CRES 304 7
1 -9 3/4 O.D. x .049 WALL TUBE HAYNES 6
417246
COPY A
AEROJET GOVERNMENT
APPLICABLE SPECIFICATIONS
1 -7 3/4 DIA BAR 5
REMOVED 4
REMOVED 3
1 -5 3/4 O.D. x .049 WALL TUBE 2
1 -3 REMOVED 1
LIST OF MATERIAL
UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES
J. JAGGER 4/3/61
TURBINE SIMULATOR - CORROSION LOOP
Aerojet-General NUCLEONICS SAN RAMON, CALIFORNIA
417246
417247 TEST
APPLICATION
5-10-61

FIGURE 7



#3 NOZZLE INLET

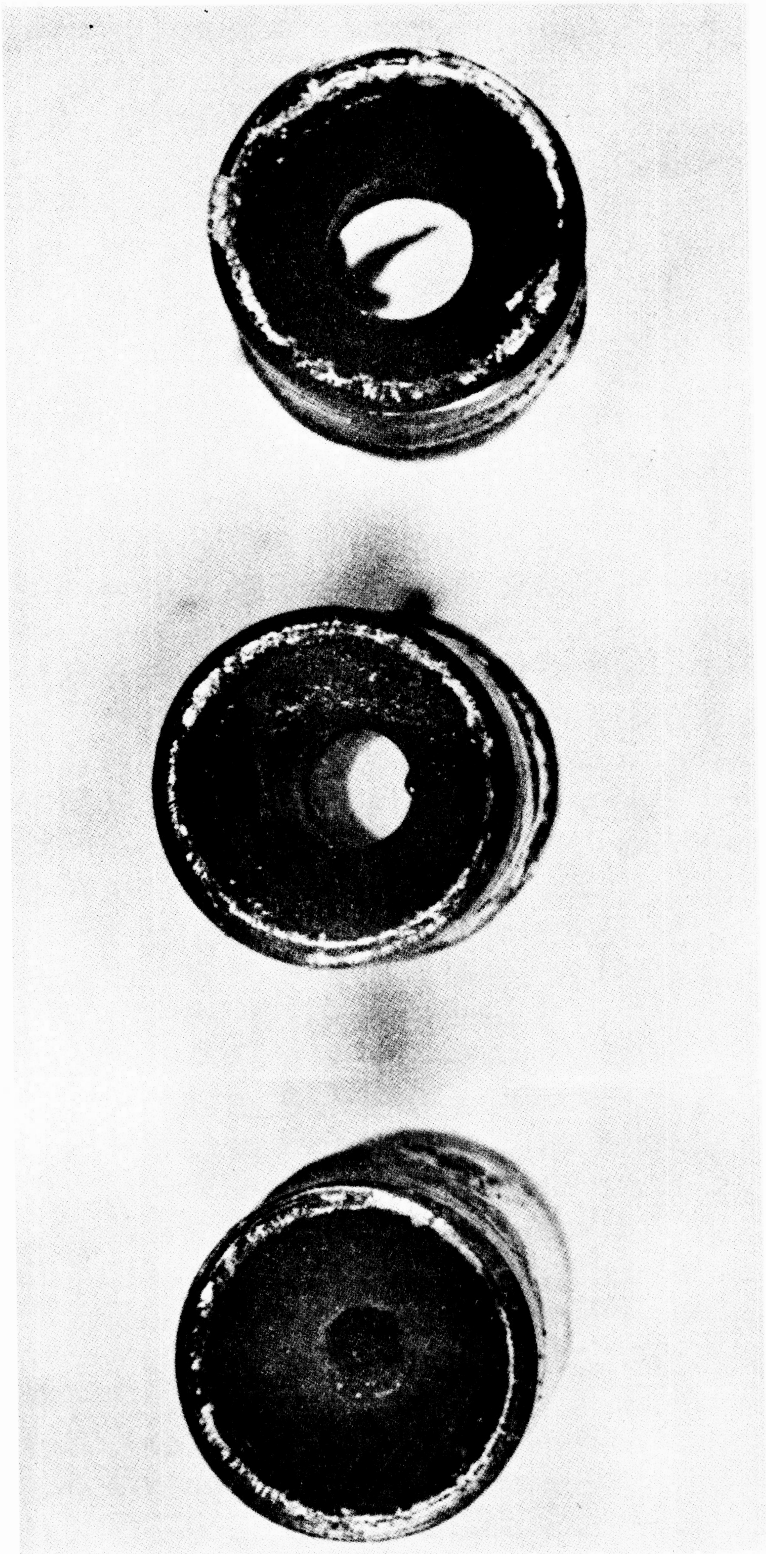
#2 NOZZLE INLET

#1 NOZZLE INLET

2X

TURBINE SIMULATOR NOZZLES (HAYNES-25)

FIGURE 8



#1 NOZZLE OUTLET

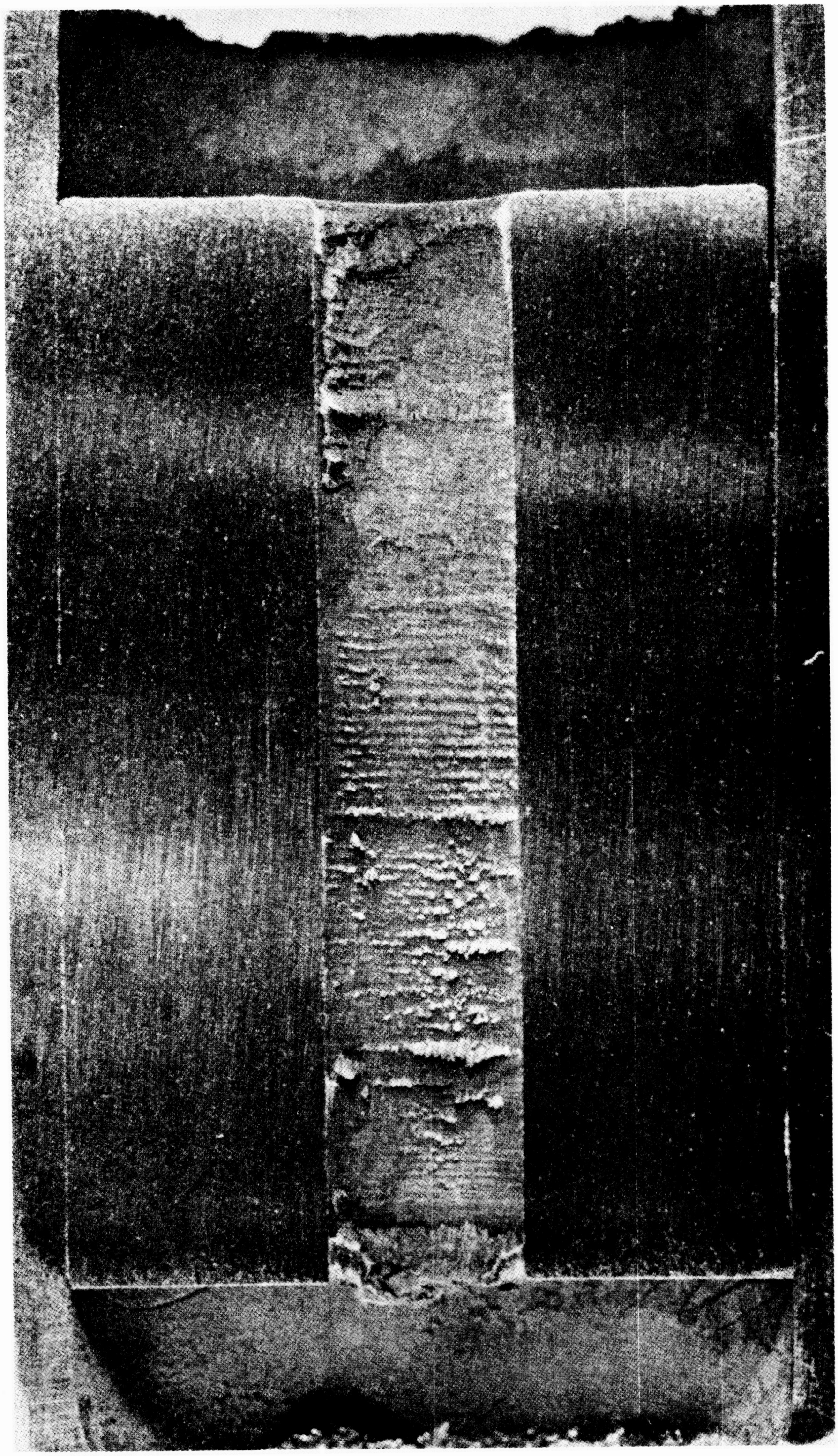
#2 NOZZLE OUTLET

#3 NOZZLE OUTLET

2X

TURBINE SIMULATOR NOZZLES (HAYNES-25)

FIGURE 9

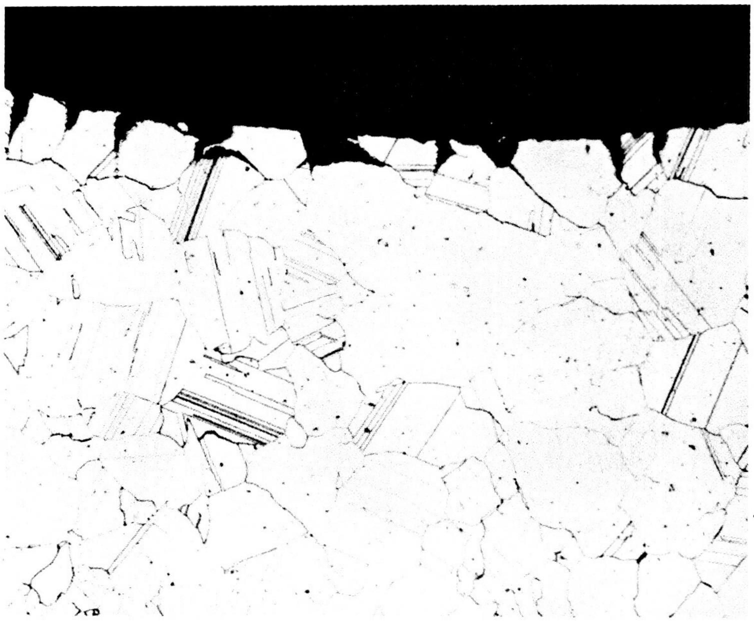


INLET ⟶ OUTLET ⟶

4X
TURBINE SIMULATOR NOZZLE #1

FIGURE 10

PHOTO MICRO GRAPHS OF HAYNES ALLOY 25 TURBINE SIMULATOR NOZZLE #1 FROM SNAP-8 CORROSION LOOP #1

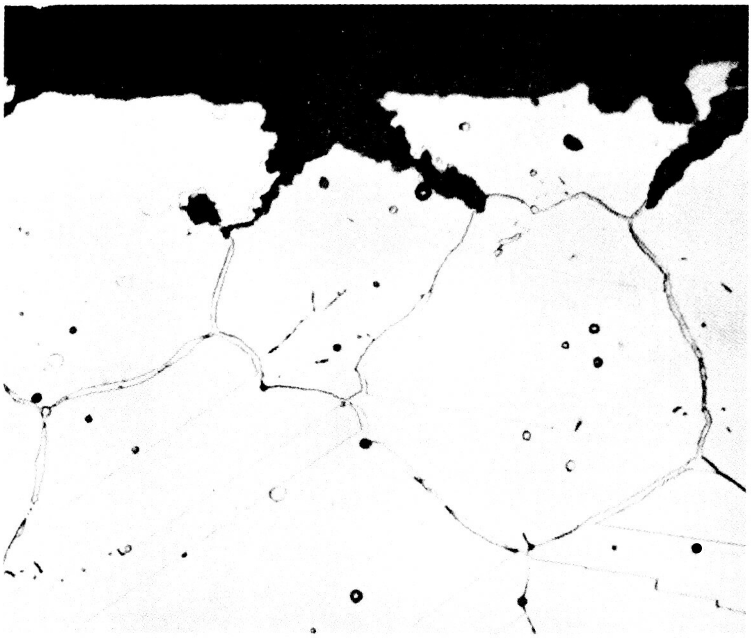


INSIDE ORIFICE

NOTE: ERODED SURFACE

2443 100X

ETCHED: GLYCERA REGIA



INSIDE ORIFICE

NOTE: ERODED SURFACE AND UNKNOWN PHASE FORMATION AT GRAIN BOUNDARY

2442 500X

ETCHED: GLYCERA REGIA

FIGURE 11

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